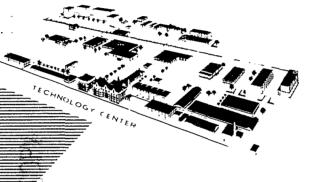
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IMPROVED VANADIUM-BASE ALLOYS

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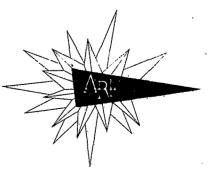
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(b) IMPROVED VANADIUM-BASE ALLOYS.

(2) Contract NOw 61-0417-c
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(7) (Bimonthly Repert. No. 39, 1 apr - 31 yray 61.

April 1, 1961 - May 31, 1961

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Department of the Navy Bureau of Naval Weapons Washington 25, D. C.

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## IMPROVED VANADIUM-BASE ALLOYS

# ABSTRACT

Promising V-Cb alloys were produced and fabricated to sheet by hot or cold working techniques. Excellent workability was evidenced by the fact that most of the compositions could be rolled to 0.050 inch sheet.

Metallographic and hardness studies on the sheet materials indicated that boron and carbon added to V-1Ti-60Cb raises the recrystallization temperature 200 to 400 F. Short-time stress-rupture properties were determined for several alloys at 2000 F; the alloy V-5Ti-20Cb-0.05C failed after 22 hours at a stress of 10,000 psi. A 50-pound ingot of V-5Ti-20Cb was double arc-melted and extruded to 3-inch diameter bar by a commercial vendor. Externally the bar appeared to be sound.

#### IMPROVED VANADIUM-BASE ALLOYS

## I. INTRODUCTION

This is the third bimonthly progress report on the subject program and covers the interval April 1 to May 31, 1961. Vanadium-columbium alloys are being investigated since they exhibit an excellent combination of fabricability, weldability, and elevated temperature strength. Most of the work during this interval was devoted to the preparation of additional sheet materials based on V-5Ti-20Cb and V-60Cb. Recrystallization temperatures of the new compositions were determined by metallographic and hardness studies of annealed sheet. Stress-rupture properties at 2000°F and tensile data at room temperature, 2000°, and 2200°F were obtained. In addition, a 50-pound 6-inch diameter ingot of V-5Ti-20Cb was double consumable-arc melted and extruded to 3-inch diameter bar by a commercial vendor.

#### II. DISCUSSION OF RESULTS

#### A. Alloys Under Investigation

Table I lists the alloys studied during this reporting interval.

Nonconsumable-electrode arc-melting techniques were used to prepare 200-gram ingots. As can be noted, the majority of these compositions are either V-5Ti-20Cb or V-60Cb base materials complexed with one or two other alloying agents. These alloys were selected to evaluate the effects of dispersion-producing or solid-solution additions on elevated-temperature strength properties. Small nitrogen and oxygen additions were included in this group. Previous results indicated that materials prepared using lower purity vanadium were stronger at elevated temperatures than were similar materials made with higher purity vanadium. Principal differences observed in the vanadium purity were in the oxygen and nitrogen content.

The remaining compositions were selected to further study materials which exhibited promise for 1200° to 1400°F service. In previous studies, 20% titanium proved to be the best strengthener at 1200°F; therefore, it was \* All compositions are reported in weight per cent.

included in all these compositions. Not listed in Table I is the 50-pound V-5Ti-20Cb alloy ingot currently being processed by Universal-Cyclops Corporation. This ingot was double consumable-electrode arc-melted to a final diameter of 6 inches. After double melting, the ingot weighed approximately 45 pounds; subsequent scalping to remove the crown and reduce the diameter gave a yield of 35 lbs.

#### B. Ingot Fabrication

The fabrication procedures employed and the fabrication results are also noted in Table I; sheet of excellent quality was obtained for the majority of the compositions. The only ingots which could not be fabricated by either hot or cold working were the V-20Ti-10Cb-15Cr and V-20Ti-10Cb-10Al alloys. In fact, both of these compositions were exceedingly hard (over 400 VPN) and exhibited thermal cracking after melting.

Similar cold working techniques were attempted on all the ingots except those containing chromium, silicon, or aluminum. The advisability of this procedure became questionable since the alloys containing more than 0.025% oxygen or carbon developed small edge cracks after being cold worked to approximately a 5% reduction. Subsequent annealing and cold working caused the formation of additional edge cracks. Therefore, it was decided that the best yield could be obtained by employing hot and cold working procedures. Ingots were encapsulated in stainless steel, and hot worked at 2400°F to approximately a 50% reduction, after which the stainless steel was removed and cold rolling was resumed. All compositions were cold rolled at least 50%.

Besides the small ingot fabrication described above, the 50-pound V-5Ti-20Cb alloy ingot was extruded to 3-inch diameter bar by Universal-Cyclops Corporation. Prior to extrusion the ingot was encapsulated in mild steel and heated in molten barium chloride salt to 2300°F. Extrusion was then performed at approximately 2200°F under pressures of 54 to 75 tsi. Externally, the extruded bar appeared to be sound. Universal-Cyclops Corporation will retain approximately one third of the extruded bar for fabrication to 0.050 inch sheet; the remainder will be processed to sheet at the Foundation.

## C. Recrystallization and Hardness Studies

Specimens for microexamination were prepared from each sheet alloy in order to determine the effect of the various additives upon the recrystallization temperature of the basic compositions. The samples were encapsulated in Vycor or quartz and annealed at 1800°, 2000°, 2400°, and 2600° F for 1/2 hour, then water quenched. Microexamination revealed that an annealing temperature of 2000°F was required for complete recrystallization of the solid solution V-60Cb alloys complexed with hafnium, zirconium, molybdenum, or tungsten. Complexing V-1Ti-60Cb with oxygen, nitrogen, or carbon raised the recrystallization temperature to 2200°F, and additions of boron raised the recrystallization temperature to 2400°F. All these latter additions resulted in the presence of very fine, uniformly distributed precipitates at the recrystallization temperature. When the solid solution alloys were annealed at temperatures above the recrystallization temperature, some grain growth occurred. Annealing the dispersion-hardened alloys above the recrystallization range caused some precipitates to enter into solution. The V-5Ti-20Cb alloys complexed with nitrogen or oxygen also had visible precipitates at all annealing temperatures; however, these complexed alloys recrystallized at 2000°F.

It was observed from the hardness curves that all the materials exhibited a minimum hardness at 200°F below the recrystallization temperature. Annealing at higher temperatures caused increased solution of precipitates or inclusions which accounted for the increased hardness. Also it was noticed that alloys containing precipitates visible at 1000 magnification were harder at all annealing temperatures than were the basic compositions. In the case of nitrogen or oxygen, this displacement in hardness amounted to as much as 20 VPN; for carbon and boron, hardness rises were approximately 15 VPN.

Metallographic and hardness studies were also conducted on the age-hardenable silicon containing compositions. Microexamination of these materials revealed that an annealing temperature of 2200°F is required for complete recrystallization. The usefulness of these alloys is questionable since an overabundance of precipitate was observed, even after annealing at 2600°F. Also, these materials exhibited relatively high hardness values even ARMOUR RESEARCH FOUNDATION OF ILLINOIS INSTITUTE OF TECHNOLOGY

in the fully annealed condition. For example, a Vickers hardness number of 310 was measured for V-20Ti-10Cb-2Si after annealing at 2200°F for 1/2 hour. Further hardening through aging would result in higher hardness and possible embrittlement. However, aging studies are in progress, and the results will be given in a subsequent report.

#### D. Tensile and Stress Rupture Test Results

Table II lists the results of tensile tests conducted at room temperature, 2000°, and 2200°F. All elevated temperature tests were performed in an inert atmosphere. Results for the V-5Ti-20Cb alloy in both the annealed and welded condition are included for comparison.

The alloy V-5Ti-20Cb was annealed at 3000°F for 1 hour to cause grain growth. Data show that a slight reduction in room-temperature ductility and elevated temperature strength resulted from the extremely large grain size. Previous studies of welded specimens of V-5Ti-20Cb also showed similar trends due to large grains in the weld and heat-affected zone.

Solid solution V-60Cb, V-0.5Ti-60Cb and V-1Ti-60Cb were tensile tested at temperatures up to 2200°F. Data from Table II show that each of these compositions suffers a drastic reduction in strength at 2200°F. Compared to V-5Ti-20Cb which had tensile elongations of less than 20%, the V-Ti-60Cb materials had excessively high elongations (86 to 140 per cent) at 2000° and 2200°F. These results do not represent optimum properties, as further complexing can certainly be expected to yield improved strength. Some losses in ductility which will occur by complexing can also be tolerated since these compositions have more than adequate tensile elongation (over 20% at room temperature). Fortunately V-1Ti-60Cb had been selected for further complexing. Of the V-Ti-60Cb alloys, this one has high room-temperature ductility (21%) and is the strongest at 2200°F.

Several vanadium-base alloys were tested for short-time stress-rupture strength at 2000°F. A stress of 10,000 psi was applied to all specimens. At this stress level the V-5Ti-20Cb-0.03C alloy failed in 18 hours and V-5Ti-20Cb-0.05C failed in 22 hours. These results indicate a significant improvement over the basic V-5Ti-20Cb composition which failed in 4 1/2 hours at the same level, and a moderate improvement over the alloy V-5Ti-20Cb-0.25C which failed in 20 hours under a stress of 8,000 psi.

# III. SUMMARY

Promising vanadium-columbium base alloys for 1200° to 1400°F, and 1800° to 2200°F applications were prepared. The moderate temperature V-Cb alloys contained 20% titanium with chromium, aluminum, or silicon additions; elevated temperature alloys consisted of V-5Ti-20Cb or V-60Cb base compositions complexed with boron, carbon, oxygen, or nitrogen. The majority of these materials were rendered to excellent 0.050 inch sheet by hot or cold rolling techniques. In addition, a 50-pound V-5Ti-20Cb ingot was double arc-melted and extruded to 3-inch diameter bar by a commercial vendor.

Metallographic and hardness studies were completed on all materials rolled to sheet. Silicon and carbon raised the recrystallization temperature to 2200°F, and boron raised the recrystallization temperature to 2400°F. Minimum hardness was measured on all materials at 200°F below the recrystallization temperature. Higher annealing temperatures caused precipitates or inclusions to enter into solution, resulting in increased hardness.

Tensile evaluations conducted at 2200°F indicate that V-1Ti-60Cb, with an ultimate tensile strength of 38,000 psi, is the strongest of the V-60Cb solid solution alloys. Stress-rupture data at 2000°F indicate that improvement in longer time properties can be realized by complexing with carbon. The V-5Ti-20Cb-0.05C withstood a stress of 10,000 psi for 22 hours at 2000°F compared to failure in 4 1/2 hours for the V-5Ti-20Cb base under a similar load.

#### IV. FUTURE WORK

Room-temperature and elevated temperature testing will continue on the materials outlined in this report. Although the majority of compositions will be evaluated at 2000° and 2200°F, a few alloys for lower-temperature applications will be screened at 1200°F. Selection of additional compositions will be based upon results obtained on the materials in process. The 50-pound 3-inch diameter extruded bar of V-5Ti-20Cb will be fabricated to sheet at the Foundation and also by a commercial vendor. Material

obtained will be evaluated for mechanical properties, and also some sheet will be supplied to other agencies for liquid metal corrosion studies.

## V. CONTRIBUTING PERSONNEL AND LOGBOOKS

The following personnel contributed to the work reported herein:

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B. R. Rajala

Project Leader

L. B. Richard

Project Technician

R. J. Van Thyne

Advisor

The data accumulated on this project may be found in Foundation Logbooks C-10872 and C-10873.

Respectfully submitted,

ARMOUR RESEARCH FOUNDATION OF ILLINOIS INSTITUTE OF TECHNOLOGY

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TABLE I

VANADIUM ALLOYS PREPARED AND IN PROCESS

Composition (w/o)	Working Process	Fabrication Results
1Ti-60Cb-0.025C	CR	Excellent sheet
1Ti-60Cb-0.05C	HR	Fair sheet, some edge cracking
1Ti-60Cb-0.025B	HR	Excellent sheet
fTi-60Cb-0.05B	HR	Excellent sheet
1Ti-60Cb-0.025C-0.025B	HR	Excellent sheet
0.5Zr-60Cb	CR	Excellent sheet
1.0Zr-60Cb	CR	Excellent sheet
2.0Zr-60Cb	CR	Excellent sheet
0.5Hf-60Cb	CR	Excellent sheet
1.0Hf-60Cb	CR	Excellent sheet
2.0Hf-60Cb	CR	Excellent sheet
1Ti-60Cb-3.0Mo	CR	Excellent sheet
1Ti-60Cb-5.0W	CR	Excellent sheet
1Ti-60Cb-0.025N	CR	Fair sheet - some edge cracking
1Ti-60Cb-0.05N	HR	Fair sheet - some large edge cracks
1Ti-60Cb-0.025O	CR	Excellent sheet
fTi-60Cb-0.05O	HR	Fair sheet - some edge cracking
5Ti-20Cb-0.025O	HR	Excellent sheet
5Ti-20Cb-0.05O	HR	Excellent sheet
5Ti-20Cb-0.07O	HR	Fair sheet - some edge cracking
5Ti-20Cb-0.05O-0.025C	HR	Fair sheet - some edge cracking
5Ti-20Cb-0.05O-0.05C	HR	Marginal sheet - some surface defects

## TABLE I (continued)

Composition (w/o)	Working Process	Fabrication Results
20Ti-10Cb-2Si	HR	Excellent sheet
20Ti-20Cb-2Si	HR	Excellent sheet
20Ti-40Cb-2Si	HR	Excellent sheet
20Ti-10Cb-15Cr	Ņot worked	Thermal cracks, after melting
20Ti-10Cb-10Al	Not worked	Thermal cracks, after melting
20Ti-20Cr	HR	Fair sheet - some edge cracking
20Ti-10Cr	HR	Excellent sheet

CR - Cold rolled 15%, annealed at 2000°F for 1/2 hr--cold rolled to 50 mil sheet.

HR - Hot rolled at 2400°F to a 50% reduction, followed by cold rolling.

TABLE II

TENSILE PROPERTIES OF VANADIUM-BASE ALLOYS

	Room	Temperature	9		2000° F		2	2200° F	
	Ultimate			Ultimate			Ultimate		
	Tensile	Yield		Tensile	Yield		Tensile	Yield	
	$\operatorname{Strength}$	$\operatorname{Str}$ ength		Strength	Strength		Strength	Strength	
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(w/o)	psi)	psi)	(%)	psi")	psi)	(%)	psi)	(isd	(%)
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V-5Ti-20Cb	105.8	85.6	20	47.1	42. 1	2.4	1	1	!
70				•	•	1			
V-5Ti-20Cb	104.8	87.8	16	42.1	41.7	ø	!	1 1	1
O :	1								
V -60Cb	147.5	130.1	21	57.3	52.4	98	33.1	28.1	140
V-0.5Ti-60Ch <sup>C</sup>	152 0	;	23	0 07	u 7	0	2 2 2	1	7
) ) ) ()	9		Ç	00.7	04.0	107	22.2	7.67	140
V-1.0Ti-60Cb	151.0	129.7	21	59.	53.4	87	37.8	32.3	114
							•	) ; }	1

a. Annealed at 2000° F for 1/2 hour - furnace cooled.
b. Annealed at 2000° F for 1/2 hour - water quenched.
c. Annealed at 2200° F for 1/2 hour - water quenched.
d. Annealed and welded.